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The Investigation of Fabrication Processing for Lotus-type Porous Magnesium by the In-situ Reaction and Unidirectional Solidification Method

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Abstract

In this paper, a novel safe and high-efficient fabrication processing named in-situ reaction and unidirectional solidification method was presented for lotus-type porous metals. The fabrication process of lotus-type porous magnesium by the in-situ reaction method had been investigated, and the rule of effect of processing parameters on pore structure and porosity was analyzed. The results showed that the porosity of the samples fabricated under different processes conditions had a wide range from 2.4 percent to 54.2 percent. The content of water, the addition of powder, the solidification rate, and casting temperature all had significant effect on the porosity and pore structure. The porosity of the samples increased with increase of the content of water, addition of powder or the solidification rate. But increasing the casting temperature would make the porosity of samples first increase then decrease. The pore in sample fabricated by in-situ reaction and unidirectional solidification method was round and regular, pore wall was very smooth, distribution of pore size was relatively uniform, and the pore growth direction was parallel to solidification.

Keywords: In-situ reaction method; lotus-type magnesium; porosity; unidirectional solidification

1. Introduction

Porous metals are new engineering materials with lower density, higher specific strength, larger surface area, good permeability and biological compatibility compared with dense metal, and have been used as important materials for structural light-weight part, heat-exchanger, filter, sound absorption, bioengineering etc [1, 2]. In recent years, lotus-type porous metal, as a new kind of porous metal, has been substantially developed [3]. Compared with foam metal, its pore structure is long and straight

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cylindrical pores arrayed along a certain direction in metal matrix instead of sphere or polygon pores randomly distributed in metallic matrix. Such lotus-type porous metal has more excellent mechanical properties, such as no obvious stress concentration, high tensile strength, high compressive strength and high stress platform etc [4, 5]. Because of widespread promising application for light-weight structural part, power-absorbing, and special filter etc due to unique structure and excellent properties, lotus-type porous metals have caused extensive research interest [6, 7]. Several fabrication methods such as gas-solid eutectic unidirectional solidification process, continuous zone melting method, and continuous casting technique, have been developed in recent years [8-10]. However, the problems of poor safety, complicated operation, low production efficiency and unsuitability for large scale production and high cost in present fabrication processes, have restricted the large-scale application of lotus-type porous metal. Therefore, the investigation and development on novel safe and high-efficient fabrication processing has great significance to the application of lotus type metal.

In this work, a novel safe and high-efficient fabrication processing named in-situ reaction and unidirectional solidification method was presented and the fabrication of lotus-type porous magnesium by this method had been investigated. The density and porosity and pore morphology of the samples had been characterized and the rule of effect of processing parameters on pore structure and porosity was analyzed.

2. Experimental

2.1. Experimental principle

The principle of fabricating lotus-type porous metals by the in-situ reaction and unidirectional solidification method was illustrated in Fig. 1. The fabricating process of a lotus type metal was as follows: First, metal was melted in crucible under the atmosphere. Then, the molten metal was cast in a mould with some hydrous coating covered on its inner surface and a water-cooling copper crystallizer on its bottom, and reacted in-situ to the water in the hydrous coating during which hydrogen generated and dissolved in molten metal. And then, due to the cooling of the crystallizer, the liquid metal in the mould started to solidify along unidirection from the bottom of the mould, and at the same time hydrogen gas was liberated because of its different solubility in solid and liquid metal. Thus, the cylindrical pores formed and grew along with solidification of liquid metal and unidirectional growth of metal matrix, and at last lotus type metal was fabricated. In order to decrease the radial conduction of heat and enhance the axial conduction of heat to ensure the direction of solidification along axial direction, the mould was made of ceramics material with low thermal conductivity.

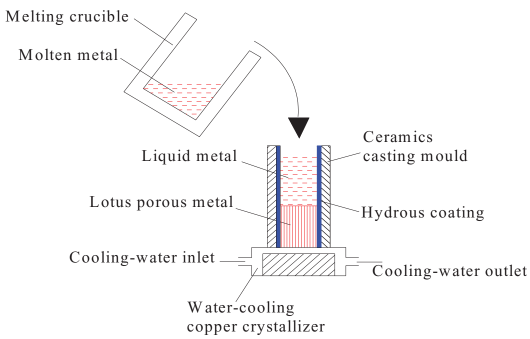

Fig. 1. Schematic diagram of in-situ reaction and unidirectional solidification processing for fabricating lotus porous metal.

2.2. Fabrication of lotus porous magnesium

In this work, 99.9% pure magnesium was used. Lotus type porous magnesium was fabricated by self-made device. Magnesium ingot was melt in a graphite crucible using an electrical resistance furnace; the temperature was measured by the nickel chromium-nickel silicon thermocouple with protection tube. The Al_2O_3 ceramics casting mould with an inner diameter of 50mm and a wall thickness of 4mm as well as a height of 150mm was adopted. T2 copper was used for making water-cooling copper crystallizer.

First, some volume of sodium silicate binder was taken by measuring cylinder (M=2), and some alumina powder with an average diameter of 50μm was weighed by an analytical balance. Then, binder and powder were uniformly mixed to be coating material in a beaker, and coating was uniformly spread on the inner surface of ceramics casting mould. And then, the casting mould was fixed on the water-cooling copper crystallizer. Second, magnesium ingot was melt in graphite crucible, and the molten temperature was set by an automatical temperature controller. When the temperature was increased to the set value, molten magnesium was cast into the pre-coated alumina casting mold through funnel. The lotus-type porous magnesium was finally formed in the way of unidirectional solidification. Finally, the porous ingot was taken out from the mould and prepared for analysis. In this experiment, effect of processing parameters on pore structure and porosity was investigated by varying solidification velocity, addition of sodium silicate binder, casting temperature, addition of powder.

2.3. Characterization of lotus porous magnesium

After both the top and bottom of the lotus porous magnesium billet were removed, the samples were incised along longitudinal and transverse sections for observing porous morphology. The weight of porous sample was weighed by an analytical balance and its volume was calculated by its sizes, then porosity (P) of the samples can be calculated by the following Eq. (1),

$$P = \left(1 - \frac{M/V}{\rho_d} \right) \times 100\% \tag{1}$$

Where M is weight of lotus-type porous magnesium (g), V is volume of lotus-type porous magnesium (cm^3); ρ_d is density of dense pure magnesium (g/cm^3).

3. Results and Discussion

3.1. Effect of addition of sodium silicate binder

In this experiment, effect of addition of sodium silicate binder on porosity and morphology of the lotus porous magnesium was investigated by a single variation of addition of sodium silicate binder. The detailed processing parameters used in the preparation experiment and the corresponding results were tabulated in Table 1, and the typical porous structure morphologies of lotus porous magnesium were shown in Fig. 2.

Table 1. Effect of the addition of sodium silicate binder on density and porosity of samples.

Sample No.	Na_2SiO_3 addition (ml)	Density (g/cm^3)	Porosity (%)	Other parameters
1	3	1.696	2.43	The addition of Al_2O_3 powder
2	5	1.289	25.84	was 4g, and the casting
3	7	1.251	28.05	temperature was 760℃
4	9	0.797	54.15	

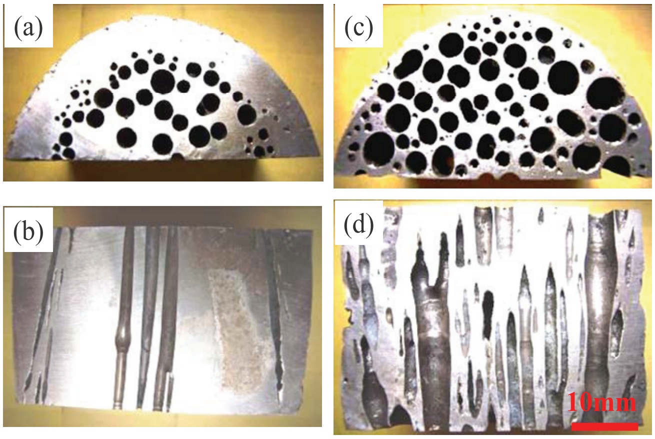

Fig. 2. Effect of the addition of sodium silicate binder on pore morphology of transverse and longitudinal section of sample: (a) and (b) 5 ml, (c) and (d) 9 ml.

It can be seen from Table 1 that the porosity of the sample increases with increment of addition of sodium silicate binder. The main reason was that the amount of hydrogen gas generated in the in-situ reaction between molten magnesium and water in the hydrous coating had influenced the porosity of sample. Less the addition of sodium silicate binder was (e.g. 5ml), less the amount of hydrogen gas produced by the in-situ reaction was, and harder hydrogen dissolved into molten magnesium was to be saturated, thus, less pores could be generated. However, because with solidification progressing, the concentration of hydrogen in solidification front would be enriched gradually, hydrogen dissolved into molten magnesium would be saturated and pores would form at last when the interface of solidification had a distance from the bottom of the mould (about 11mm). When the addition of sodium silicate binder increased to a value (e.g. 9ml), molten magnesium could reacted with water acutely and a plenty of hydrogen gas was rapidly produced and dissolved into molten magnesium. Thus, hydrogen gas would become saturated rapidly and pores formed easily. Therefore, a billet with more pores and larger porosity was fabricated.

It could be seen from Fig. 2(a) and (c) that all samples had a pore-free zone, but its width had a little difference between the samples from different processing parameter, the more addition of sodium silicate binder was, the less the width of pore-free zone was. The reason for the formation of pore-free zone was that the radial elimination of heat had not been prevented completely, therefore, when magnesium liquid solidified from the bottom of mould to top, a very slow solidification from side wall to center of the mould would happen and a pore-free zone form. Because the formation of pores needed a course of nucleation and growth, the first solidified superficial shell had no pores. Similarly, due to the effect of the amount of hydrogen gas generated in the in-situ reaction, the more addition of sodium silicate binder was, the more hydrogen gas generated, and the more easily pores nucleated and grew. So the more addition of sodium silicate binder was, the less the width of pore-free zone was.

It could be also observed from Fig. 2(b) and (d) that some pores were through, others were closed, but their walls were all smooth. It was also seen especially obviously from Fig. 2(b) that the direction of the pores near the lateral surface of the billet was not parallel to the axial direction of the billet, but had an angle with it. The growth direction of pores was determined by that of heat transmission, therefore when a little radial elimination of heat near the inner face of the mould had not been prevented completely in this experiment, the resultant direction of heat transmission near the inner face of mould had a small angle with the axial direction. Certainly, because the radial elimination of heat had little effect on heat transfer in the centre of mould, the pores in centre were parallel to axial direction of the billet.

3.2. Effect of addition of powder

In this experiment, effect of addition of powder on porosity and morphology of the lotus porous magnesium was investigated by a single variation of addition of powder. The detailed processing parameters used in the preparation experiment and the corresponding results were tabulated in Table 2, and the typical porous structure morphologies of lotus porous magnesium were shown in Fig. 3.

Table 2. Effect of the addition of alumina powder on density and porosity of sample.

Sample No.	Powder addition (g)	Density (g/cm^3)	Porosity (%)	Other parameters
1	2	1.418	18.39	The addition of sodium
2	4	1.289	25.84	silicate binder was 5ml,
3	6	0.949	45.38	and the casting
4	8	0.927	46.67	temperature was 760℃

Table 2 showed that the porosity of sample had been influenced by addition of powder. Before the addition of powder was up to a value, with increasing it, the porosity would be increased remarkably. However, when it was above the value, the porosity had a slight change with its increase.

The effect of addition of powder on porosity of the lotus-type porous magnesium can be discussed through analyzing nucleation mechanism of pore. Based on classical nucleation, homogeneous and inhomogeneous nucleation could happen in the formation of pores, but inhomogeneous nucleation was predominating in this course. In this experiment, during the molten magnesium was cast in the mould and solidified, due to the flow of magnesium liquid and agitated by gas, alumina powder in the coating on the inner surface of casting mould would be dispersed into the magnesium liquid and acted as the nucleus of pores. When the addition of powder was insufficient (e.g. 2 g), the nucleus for pores were too lack to overcome the nucleating resistance and form a large number of pores, so the porosity of prepared sample was small. Conversely, if the addition of powder was much (e.g. 6 g), the nucleus for pores were sufficient, for the same reason, the porosity of sample was large. However, too much addition of powder had slight effect on increase of porosity, for instance porosities at addition of powder of 8 g and 6 g had a difference of about 1.3 %. The result indicated that 6 g powder might provide enough nucleation sites for so many pores, and excessive powder might contribute little to porosity.

It could be also observed from Fig. 3 that major pores were through, and their walls were all smooth. The direction of the pores near the lateral surface of the billet was also partial to the centre of the billet.

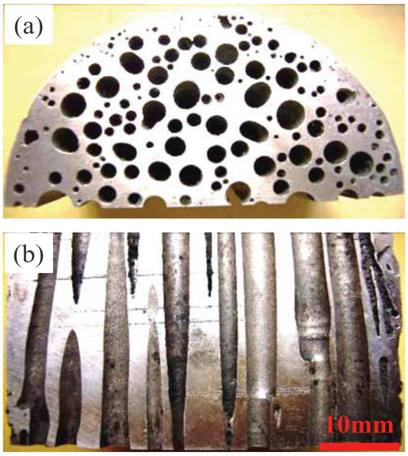

Fig. 3. Pore morphology of transverse and longitudinal section of sample under the addition of powder of 8g: (a) transverse section, (b) longitudinal section.

3.3. Effect of solidification rate

In this experiment, in order to investigate the effect of solidification rate on porosity and morphology of the lotus porous magnesium, different thickness of graphite sheets were put on the place between the bottom of mould and water-cooling copper crystallizer to change the rate of heat transfer and thus change solidification rate.

The detailed processing parameters used in the preparation experiment and the corresponding results were tabulated in Table 3, and the typical porous structure morphologies of lotus porous magnesium were shown in Fig. 4.

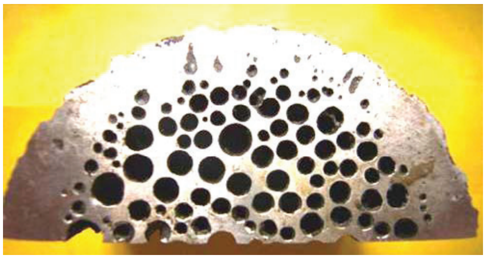

Fig. 4. Pore morphology of transverse section of the sample under the graphite sheet thickness of 1mm.

Table 3. Effect of solidification rate on density and porosity of sample.

Sample No.	Graphite sheet thickness (mm)	Density (g/cm^3)	Porosity (%)	Other parameters
1	0	0.949	45.38	The addition of sodium silicate binder
2	1	1.083	37.68	was 5ml, the addition of alumina powder
3	2	1.381	20.55	was 6g, the casting temperature was 760℃

The results in Table 3 showed that the porosity of sample increased with increasing the solidification rate. Compared with nucleation and growth of metal, that of pores was a special course. On the one hand, with solidification proceeding, supersaturated hydrogen would be liberated on the solid-liquid interface of metal, which would result in the nucleation and growth of pores in metal matrix, on the other hand, the liberated hydrogen could also float and escape from magnesium liquid and not involve in the formation of pores. So it was not hard to understand that with increasing the solidification rate, degree of supersaturation of hydrogen would increase on the solid-liquid interface of metal, which would accelerate the nucleation of pores, thus the porosity would increase. Furthermore, when solidification rate was fast, the hydrogen was not easy to float and escape from magnesium liquid, which was also in favor of the increase of porosity.

3.4. Effect of casting temperature

In this experiment, effect of casting temperature on porosity and morphology of the lotus porous magnesium was investigated by a single variation of casting temperature. The detailed processing parameters used in the preparation experiment and the corresponding results were tabulated in Table 4, and the typical porous structure morphologies of lotus porous magnesium were shown in Fig. 5.

Table 4. Effect of casting temperature on density and porosity of sample

Sample No.	Powder addition (g)	Density (g/cm^3)	Porosity (%)	Other parameters
1	680	1.692	2.64	The addition of sodium silicate
2	720	1.054	39.33	binder was 9ml, the addition of
3	760	0.797	54.15	alumina powder was 4g ,
4	800	0.813	53.21	and without graphite sheet

It could be seen from the variation of porosity with casting temperature in the Table 4 that the porosity first increased with increasing the casting temperature, and decreased slightly above 760℃. When casting temperature was too low (e.g. 680℃), on the one hand, molten magnesium in mould would be solidified too rapidly, and had no adequate time to react with water in coating, and at the same time, it has no adequate time for hydrogen to dissolve in it. On the other hand, the solubility of hydrogen in molten magnesium increased with the increase of the casting temperature. So, the hydrogen dissolved in the molten magnesium was little and that liberated during solidification was even less. Due to the above two reasons, at a low casting temperature, the porosity of sample was small. Conversely, when the casting temperature was increased, the reaction would be adequate and the hydrogen dissolved in molten magnesium also increased remarkably, so the porosity of sample would increase correspondingly.

However, if the casting temperature was too high (e.g. 800℃), the solidified time for molten magnesium would become too long, and the hydrogen escaped from molten magnesium to atmosphere would increase. Thus, the porosity decreased slightly at a too high casting temperature.

It could be also observed from Fig. 5 that pores were straight, and their walls were all smooth and their diameters were uniform.

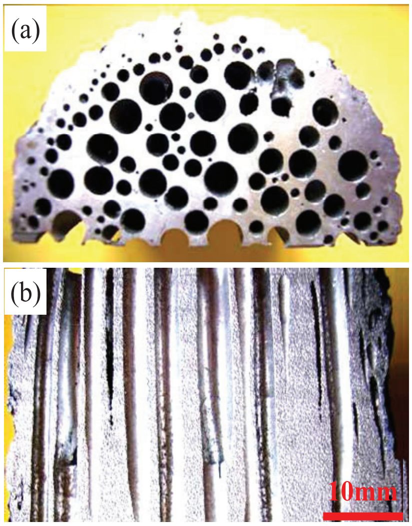

Fig. 5. Pore morphology of transverse and longitudinal section of sample under the casting temperature of 800℃: (a) transverse section, (b) longitudinal section.

4. Conclusions

(1) Lotus porous magnesium with a high porosity of about 54.15 percent and a low density of 0.797 g/cm^3 was fabricated by the in-situ reaction method.

(2) The pores in sample were round and regular, pore wall was very smooth, distribution of pore size was relatively uniform, and the pore was parallel to solidification.

(3) The porosity of the samples fabricated under different processes conditions had a wide range from 2.4 percent to 54.2 percent. The addition of sodium silicate binder, the addition of powder, the solidification rate, and casting temperature all had significant effect on the porosity and pore structure.

(4) The porosity of the samples increased with increase of the addition of sodium silicate binder, addition of powder or the solidification rate. But increasing the casting temperature would make the porosity of samples first increase then decrease.

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